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Title: Method for a Countercurrent Washing of Finely Dispersed Crude Terephthalic Acid

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9109 Eagle Hills Dr . Las Vegas NV 89134 . ph: 866-722-7990 (toll-free) . fax: 702-973-6009
email: sales@tls-translations.com

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(72) Inventor:
Vaillancourt, Joseph Anthony, Wilmington, DE 19808 USA

(71) Applicant:
Hercofina, Wilmington, NC, USA

(74) Representative:
Schönwald, K., Ph.D. Engineering, 5000 Cologne, Eishold, K., certified engineer, Ph.D. Engineering, 6232 Bad Soden; Fues J., certified chemist, Ph.D.; von Kreisler, A., certified chemist; Keller, J., certified chemist; Seiting, G., certified engineer: Werner, H., certified chemist, Ph.D., Patent Attorneys, 5000 Cologne

(54) Method for a Countercurrent Washing of Finely Dispersed Crude Terephthalic Acid

A method is disclosed for a countercurrent washing of finely dispersed crude terephthalic acid with a wash liquid of significantly different density in a scrubber. In order to avoid channeling or back-mixing of the liquid and to intensify washing or scrubbing of the particles, the column is divided in the transverse direction by at least one false bottom with a number of specially dimensioned holes that influence particle penetrability. In addition, the method comprises a continuous and hydraulic sequence of steps, in which settling (or rising) occurs in one step under the influence of gravity for a period long enough for the terephthalic acid particles to collect, agglomerate and form a non-fluidized bed in the vicinity of the false bottom; a step of transport of the terephthalic acid particles with a cocurrent liquid stream then takes place, which occurs at a rate and for a period sufficient to fluidize the bed and transport the fluidized particles of terephthalic acid through the holes of the false bottom.

VON KREISLER SCHÖNWALD EISHOLD FUES
VON KREISLER KELLER SELTING WERNER

Hercules Incorporated
Wilmington, Delaware / USA

D-5000 COLOGNE 1 17 July 1981
AVK/GF/be

Claims

1. A method for a countercurrent washing of finely dispersed crude terephthalic acid with a wash liquid of significantly different density, in which the denser material is introduced to the headspace of a vertical tower containing the wash liquid, the denser material reaching the bottom of the tower is withdrawn from the tower, the other material in the bottom space of the tower is introduced to the tower and the other material reaching the top of the tower is taken off, characterized by the fact that the tower is divided in the transverse direction by at least one false bottom having a number of holes, each hole being dimensioned so that, if a non-fluidized bed of terephthalic acid particles is present in the vicinity of the false bottom, only a minority of the particles, if any, pass through the hole, but that free transport of the particles occurs through the hole, if a cocurrent liquid stream is present with a rate sufficient to fluidize the bed, and that the method of operation of the tower includes a sequence of continuous and hydraulic working steps, in which countercurrent flow of the liquid occurs through the holes in at least one step, but during which settling (or rising) under the influence of gravity occurs in one step, so that the terephthalic acid particles collect, agglomerate and form a non-fluidized bed in the vicinity of the false bottom by settling (or rising) under the influence of gravity, and during which a step of transport of the terephthalic acid particles by means of a cocurrent liquid stream then occurs at a rate and for a period sufficient to fluidize the bed and transport the fluidized terephthalic acid particles by the liquid through the holes in the false bottom.

2. The method according to Claim 1, characterized by the fact that the tower contains a number of false bottoms at vertical spacings from each other.

3. The method according to Claim 1 and 2, characterized by the fact that the sequence of working steps includes the following steps: (1) initiation and maintenance of settling (or rising) of the terephthalic acid particles under the influence of gravity for a period sufficient for the particles to collect, agglomerate and form a non-fluidized beds in the vicinity of the false bottoms; (2) initiation and maintenance of a countercurrent of liquid through the holes at a rate and for a period sufficient to fluidize the beds and expel the liquid from them; (3) initiation and maintenance of settling (or rising) of the terephthalic acid particles under the influence of gravity for a period sufficient for the particles to collect again, agglomerate and form a non-fluidized beds in the vicinity of the false bottoms under the influence of gravity by settling (or rising); and (4) initiation and maintenance of a cocurrent stream of liquid through the holes at a rate and for a period sufficient for the beds to be fluidized and the fluidized particles to be transported through the holes of the false bottoms.

4. The method according to Claim 3, characterized by the fact that the sequence of working steps is produced [by] a hydraulically and continuously controlled and regulated introduction of liquid into both end spaces of the tower and withdrawal of the liquid from both ends of the tower.

5. The method according to Claim 3, characterized by the fact that step (2) is conducted, so that the wash liquid is introduced to the end space of the tower lying opposite the end space into which the terephthalic acid particles are introduced, and that, at the same time, from the latter end space, the liquid reaching this space is withdrawn, and that step (4) is conducted, so that the liquid is taken off from the end of the tower, from which the washed terephthalic acid particles are taken off.

6. The method according to Claim 5, characterized by the fact that, simultaneously with the removal of the liquid from the end of the tower from which the washed terephthalic acid particles are withdrawn, liquid is introduced to the opposite end space of the tower.

7. The method according to Claim 6, characterized by the fact that the finely dispersed particles of crude terephthalic acid and the liquid supplied to the same end space of the tower are introduced together in the form of a suspension.

8. The method according to Claim 7, characterized by the fact that the washed terephthalic acid particles and the liquid withdrawn from the same end of the tower are taken off together in the form of a suspension.

9. The method according to Claim 8, characterized by the fact that terephthalic acid is the denser material.

10. The method according to Claim 9, characterized by the fact that the suspension of finely dispersed terephthalic acid particles and a liquid is a suspension of finely dispersed terephthalic acid particles in an aqueous mother liquor, and that the liquid introduced to the bottom space of the tower is water with a degree of contamination that lies significantly below that of the aqueous mother liquor.

METHOD FOR A COUNTERCURRENT WASHING OF FINELY DISPERSED CRUDE TEREPHALIC ACID

The invention relates to a method for the purification of terephthalic acid.

Terephthalic acid is an important commercial chemical. It is used in tonnage amounts to produce polyester polymers, from which polyester fibers, films and bottles are produced. It is produced industrially from p-xylene. In an ordinary method for the production of terephthalic acid, p-xylene is oxidized directly to terephthalic acid. The oxidation step yields a reaction mixture that contains not only the acid, but also intermediates of oxidation, like para-toluic acid, 4-carboxybenzaldehyde and the like, which must not surpass certain maximum concentrations in the end product for many, if not most, applications. Consequently, it is necessary to separate terephthalic acid from the reaction mixture and subject it to additional treatment, in order to separate the remaining contaminants, at least one of which, 4-carboxybenzaldehyde, has a tendency to be occluded by the solid terephthalic acid particles. For these reasons, significant efforts have been made in the field to find and further develop means and ways to purify crude terephthalic acid.

An early development for the purification of crude terephthalic acid is the method in a vertical scrubber, see GB-PS 557,996. In this method a suspension of particles of crude terephthalic acid in acetic acid, water or another liquid having a much lower density than terephthalic acid, and in which terephthalic acid is relatively insoluble under the prevailing conditions, is introduced to the headspace of a tower containing a wash liquid, which is generally the same liquid, but in a purer state than the suspension liquid. The wash liquid is introduced to the bottom space of the tower, the liquid reaching the headspace of the tower is taken off and a suspension of wash liquid and terephthalic acid particles that reaches the bottom is removed. In this way, the liquid fed into the tower with the suspension is expelled and the terephthalic acid particles are washed by the wash liquid. The performance of this method, however, is not as good as desired, since channeling or back-mixing of the liquid occurs in the tower.

The present invention relates to an improvement of the method for the treatment of crude terephthalic acid in a vertical scrubber.

According to the method of the present invention, the tower is divided in the transverse direction by at least one false bottom with a number of holes. Each of these holes is dimensioned, so that if a non-fluidized bed of terephthalic acid particles is in the vicinity of the false bottom, only a minority of the particles, if any, settle (or rise) through the hole, but free transport of the particles through the hole occurs with a liquid stream in the general migration direction of the terephthalic acid particles through the tower, namely, with a cocurrent liquid stream and at a rate sufficient for fluidization of the bed. In addition, the method includes the hydraulic and continuous performance of a sequence of steps, in which settling (or rising, if the wash liquid is denser than the terephthalic acid) occurs in one step under the influence of gravity for a period sufficient for the solid particles to collect, agglomerate and form a non-fluidized bed in the vicinity of the false bottom, all of this by gravity settling (or rising), and in which a step of particle transport then occurs by means of a cocurrent liquid stream through the holes with a rate and for a period sufficient for the bed to be fluidized, and the fluidized terephthalic acid particles are transported by the liquid through the holes of the false bottom. In this sequence of steps, a countercurrent of liquid through the holes of the false bottom and through the particle bed adjacent to the false bottom occurs either during settling (or rising) of the particles or in a separate step. If this occurs under the influence of gravity during the settling (or rising) step, the flow rate is chosen so that the liquid is expelled from the bed, but this rate is smaller than the settling (or rising) rate of the solid particles of an expected minimal size or a selected size, if classification of the particles is to be conducted in the column. However, if this occurs as a separate step, the rate of the countercurrent can be greater, but it and the flow time are chosen so that the bed is fluidized, but the particles are transported, at most, a short distance from the bed in the countercurrent direction.

A general embodiment of the method according to the present invention includes only two steps. In this embodiment, the liquid countercurrent in the tower is produced hydraulically during at least part of, and preferably the entire time of the settling (or rising) step under the influence of gravity. The rate of the liquid countercurrent in the region of the false bottom is set at a value

and maintained at this value, which is smaller than the rate of settling (or rising) of the solid particles occurring under the influence of gravity of the expected minimal particle size or another selected particle size, if classification of the solid particles is to be conducted in the tower. In this embodiment, the liquid countercurrent is produced hydraulically by means of introducing the wash liquid into the column from the end from which the washed terephthalic acid particles are taken off and by means of removing the liquid from the opposite end of the tower. In addition, the cocurrent liquid stream in this embodiment is produced hydraulically in the tower as a result of removing the liquid from the bottom of the tower, and preferably by means of the simultaneous introduction of liquid into the headspace of the tower, if the terephthalic acid is denser than the wash liquid, and if the wash liquid is denser than terephthalic acid, by means of removing the liquid from the top of the tower and simultaneous introduction of wash liquid into the bottom space of the tower.

Another preferred general embodiment of the method according to the invention includes both of these steps, following a combination of a first step of settling (or rising) under the influence of gravity and a subsequent step of fluidization of the bed by a countercurrent. This embodiment therefore includes the hydraulic and continuous performance of the following sequence of steps:

(1) Initiation and maintenance of settling (or rising, if the wash liquid is denser than terephthalic acid) of the terephthalic acid particles under the influence of gravity for a period sufficient for the particles to collect, agglomerate and form a non-fluidized bed in the vicinity of the false bottom;

(2) Initiation and maintenance of a liquid countercurrent through the holes of the false bottom at a rate and for a period sufficient to fluidize the bed and expel the liquid and transport the particles away from it;

(3) Initiation and maintenance of settling (or rising, if the wash liquid is denser than terephthalic acid) of the solid particles under the influence of gravity for a period sufficient for the solid particles to collect again, agglomerate and form a non-fluidized bed in the vicinity of the false bottom; and

(4) Initiation and maintenance of a cocurrent liquid stream through the holes of the false bottom at a rate and for a period sufficient for the bed to be fluidized and the fluidized particles transported through the holes.

This sequence of steps is produced hydraulically and continuously by a controlled and regulated feed of liquid into both end spaces of the tower, and as a result of the removal of liquid material from both ends of the tower.

The number of holes in each horizontal false bottom can be varied over a wide range. The number of holes should be large enough to permit an overall practicable migration rate of the terephthalic acid particles and the wash liquid through the tower. On the other hand, it should not surpass the number, at which channeling of the wash liquid and noticeable back-mixing of the displaced liquid occur at a significant distance from the holes, i.e., flow of wash liquid in streams devoid of solid particles, and flow of wash liquid through some of the holes of a false bottom in the desired direction, in which part of the stream changes its direction and, together with expelled liquid, returns through other holes to the other side of the false bottom. In this respect, during the practical performance of the present invention, the false bottom, under normal conditions, operates so that undesired channeling and back-mixing of the liquid are reduced to a minimum in the tower under normal operating conditions.

In preferred embodiments of the method according to the invention, the tower is divided in the transverse direction by a number of false bottoms, provided with holes and arranged at vertical spacings from each other, so that a number of stages are formed, and the work cycle just described is conducted hydraulically and continuously at the same time in each stage. The number of stages depends on the ease, with which the contaminants present in the supplied suspension of crude terephthalic acid can be removed, the type of contaminants, their concentration, the wash liquid, the desired output, the desired end results, etc. Consequently, no general statements can be made with respect to the number of stages, except that the number of stages can easily be determined experimentally in each special situation. The vertical spacings between false bottoms likewise depends on a number of variables, including flow rates, depths of

the non-fluidized beds, etc. For this purpose, it can only be stated in general that, at the selected flow rates and times, the false bottoms should not be arranged at such a narrow spacing that during the fluidization step of the bed in countercurrent of the liquid stream, particles are transported through the holes of one false bottom back to a preceding stage, or that during the step of transport of the solid particles in cocurrent with the liquid stream, particles are transported beyond the next step. On the other hand, the false bottoms need not be separated vertically from each other so far that the total height of the column becomes impractical and, in this respect, for a stipulated vertical spacing of the false bottoms in the corresponding embodiment of the method, the rate and duration of flow is chosen, so that the countercurrent fluidizing the bed fluidizes the bed and expels the liquid and solid particles from it, without a noticeable amount of solid particles being transported back through the holes of the preceding false bottom, and that the cocurrent fluidizing the bed fluidizes the bed and transports the solid particles through the holes of the adjacent false bottom, but does not transport a noticeable amount through the holes of the subsequent false bottom.

In a preferred embodiment of the invention, the particles of crude terephthalic acid and the cocurrent liquid are fed together into the tower in the form of a suspension. The cocurrent liquid in these embodiments can be the reaction liquid (the liquid medium in which terephthalic acid is formed), the mother liquor (the liquid, from which the crude terephthalic acid is crystallized), the wash liquid (if particles of crude terephthalic acid are suspended in the liquid) and the like. In most embodiments of the invention, washed terephthalic acid particles and the wash liquid material from the tower are used in the form of a pumpable suspension. In some embodiments, however, the washed particles are taken off in a thickened state.

In some embodiments in which the finely dispersed crude terephthalic acid is fed, together with the liquid, in the form of a suspension, into the tower, liquid is continuously withdrawn from the end of the tower in the region into which the suspension is introduced. In this embodiment, the rate of withdrawal of the slurry from the tower can be large enough so as to produce a liquid cocurrent through the tower at a rate so that a significant amount of the particles of solid material are transported away during the cocurrent step in the sequence of steps over several stages, in which case the advantage of these purification stages does not apply for such particles. In this

embodiment, the rate of the liquid cocurrent stream through the tower is preferably reduced during withdrawal of the suspension from the tower, so that during this withdrawal with a selected rate, a liquid (wash liquid or, less preferred, recycled wash liquid) is fed into the region of the end of the tower from which the suspension is withdrawn.

In some embodiments of the invention, the wash liquid is denser than terephthalic acid. The finely dispersed particles of crude terephthalic acid are then accordingly fed into the bottom space of the tower and the countercurrent wash liquid is fed into the headspace of the tower.

In most embodiments of the invention, however, terephthalic acid is denser than the wash liquid. In such embodiments, the crude terephthalic acid is fed to the tower into the headspace, preferably in the form of a suspension, and the solid material reaching the bottom of the tower is taken off, preferably in the form of a suspension, and during part of the sequence of steps, wash liquid is fed into the bottom space of the tower and liquid taken off from the headspace of the column.

The procedure now considered the best method for the performance of the present invention is explained by embodiments of the process depicted in the accompanying drawings. These drawings are part of the disclosure of this description.

Fig. 1 shows a basic view, partially in the form of a diagram, of a embodiment of an apparatus for the execution of the method according to the invention, in which terephthalic acid is the denser material, this apparatus containing a scrubber 10, parts of the external design of which were left out in the depiction in order to make the internal design more apparent;

Fig. 2 shows a layout, drawn in the cross sectional plane 2-2 shown in Fig. 1, of the baffle plate structure at the outlet of the feed line for the suspension to the scrubber 10;

Fig. 3 shows an enlarged side view, drawn in cross sectional plane 1-3 depicted in Fig. 1, showing, in an enlarged individual view, a cutout of a false bottom arranged in the transverse direction in scrubber 10;

Fig. 4 shows an enlarged layout of part of a false bottom arranged in the transverse direction of scrubber 10, which was drawn through the plane 4-4 shown in Fig. 1;

Fig. 5 shows a layout of an enlarged section through the lower end space of the scrubber of Fig. 1, which was drawn as shown in Fig. 1 through the plane 5-5;

Fig. 6, 7 and 8 show flow rate-time diagrams that explain different methods of operation of the apparatus of Fig. 1 according to three different embodiments of the method according to the invention.

Fig. 1, in particular, shows an embodiment of an apparatus for the execution of a working process for the displacement of a liquid and the washing of solids according to the present invention, in which the crude terephthalic acid is denser than the wash liquid. The apparatus includes the scrubber 10, a suspension feed line 12, which discharges into the headspace of the scrubber, the wash liquid feed line 14 that discharges into the bottom of the scrubber, as well as a line 18 to take off the liquid emerging from the top of the scrubber. The apparatus also includes a timer switch 20 connected to the valves of each of these pipelines, in order to control flow through these pipelines, in agreement with the objectives of the present invention, and to regulate the flow rate in each pipeline.

The scrubber 10 generally consists of a vertical, cylindrical structure and includes a center part 22 containing separation stages, a headspace 24 and a bottom space 26.

The center part 22 containing the separation stages, in the depicted embodiment, comprises a number of cylindrical shell elements 22, stacked one on the other, which have the same inside diameter and are situated on the same vertical axis. Horizontally running false bottoms 28 are incorporated between the ends of the shell elements 27, which are connected fluid-tight to each

other. Each of these false bottoms has a number of cylindrical through-openings or holes 30. See Fig. 3 and Fig. 4. These holes have an inside diameter that is much greater than the maximum dimension of the largest particles of the solids to be washed according to the method of the present invention. The inside diameter of holes 30 is chosen so that, under normal operating conditions, without a liquid cocurrent stream or a liquid countercurrent stream, whose velocities are equal to the settling rate of terephthalic acid particles of the smallest expected particle size or surpass them, the particles of the terephthalic acid collect in the vicinity of the false bottoms 28, agglomerate and prevent each other from passing through the holes.

The head part 24 of the scrubber 10 includes a cylindrical shell element 32 of enlarged inside diameter, a connecting shell element 34 in the form of an inverted truncated cone and a head closure cover 36. The inside diameter of the cylindrical element of enlarged diameter is significantly greater than the inside diameter of the shell of the central part 22, including the separate stages. It is chosen so that under normal operating conditions, the head part of the liquid column in the scrubber 10 operates as a clarifier. In other embodiments, the inside diameter is chosen, so that under normal operating conditions, the head part of the liquid column operates as a classifier; this means it operates so that the terephthalic acid particles are separated, because of their particle size, the particles that are finer than a selected particle size being taken off with the outflowing liquid for the purpose of being returned to circulation or for other treatment. The cylindrical part with enlarged inside diameter 32 on its lower end grades into the shell part with the shape of an inverted truncated cone 32, on whose lower end the top of the cylindrical shell of the central part 22 containing the separation stage is connected. The slope of the internal wall of the shell part with the shape of an inverted truncated cone 34 is chosen so that it is greater than the angle of repose of the particles of crude terephthalic acid, so that collection of these particles on the sloped surface is avoided on this account. The closure cover 36 closes off the headspace of tower 10. It is provided with an opening 38 through which the feed line for the suspension 12 is passed, and an opening 42, on which the inlet of the discharge tube for the outflowing liquid 18 is mounted. In other embodiments, the upper cover is left out and the inlet of the discharge tube 18 is situated on the upper side wall of the cylindrical part 32 or on the bottom of an overflow channel or overflow box on the upper end of the cylindrical part 32. The present embodiment, with a cover 36, is suitable for use in those methods in which the

temperature of the supplied suspension lies above its boiling point at atmospheric pressure and the operation is conducted under a pressure sufficient to keep the liquid in the liquid state.

Within the cylindrical part with enlarged diameter 32, the feed line for the suspension 12 ends in an outlet that is arranged at a distance from the inlet of the discharge tube 11, so that a short-circuit-like direct stream of terephthalic acid particles from that outlet to this inlet is reduced to the least possible minimum. At the outlet end of the suspension feed line 12, there is a baffle plate structure 40, see Fig. 2. It consists of a horizontal disk 46, which is arranged at a short distance beneath the outlet opening of the line part 12 and fastened to the outlet part on the end of the tube 12 by means of struts 48. The spacing of this disk or this baffle plate 46 from the outlet opening of the suspension feed line 12 is chosen so that the streams of suspension flowing from the outlet opening into the liquid of tower 10 are divided as much as possible and short-circuit-like flow of the terephthalic acid particles to the inlet opening of the discharge line 18 is limited to a minimum.

The bottom part 26 of scrubber 10 consists of a vertical cylindrical part 50, which is connected on its upper end to the lower end of the shell of the central part 22 containing the separate stages, and a bottom part 42 is connected on its lower end in the form of an inverted cone. A bottom outlet 54 for withdrawal of a suspension of washed terephthalic acid particles is situated on the lower conical tip of the bottom part 52, to which the inlet end of the suspension withdrawal tube 16 is connected. The bottom part, in the form of an inverted cone 52, also contains an inlet 56, through which the feed line 14 for the wash liquid is passed. The wash liquid feed line ends within bottom part 26 in a horizontal, coaxially arranged sprinkler ring 62. On the top of the ring (see Fig. 5), there are a number of holes 64 arranged at practically the same spacing (Fig. 5), through which the wash liquid is introduced to the bottom space of the tower.

The suspension feed line 12, the wash liquid feed line 14, the suspension discharge line 16 and the liquid discharge line 18 in the scrubber unit of Fig. 1 are each equipped with a cutoff and flow control valve with the corresponding numbers 66, 68, 70 and 72. These valves are opened, closed and regulated during opening by the valve control devices 74, 76, 78 and 80, which are connected to the timer switch 20. The valve control devices in the drawing are electro-

mechanical designs that are operated electrically via wiring to the timer switch 20. Other means, for example, pneumatic devices, hydraulic devices and the like, can be used in conjunction with a timer switch 20 for operation of the valves and therefore to control and regulate the streams in and out of the scrubber 10.

In an embodiment of the method of the present invention, as depicted in Fig. 6, the sequence of steps begins by opening the control valve 66 of the suspension feed line and the corresponding valve 72 of the suspension discharge line, and closing the wash liquid inflow valve 68, as well as the valve 70 of the liquid discharge line. As a result, a supplied suspension of finely dispersed particles of crude terephthalic acid in a liquid, for example, an aqueous mother liquor, is passed through the suspension feed line 12 into the column of wash liquid, for example, water, which is contained in tower 10, and terephthalic acid particles that have reached the bottom of the wash liquid column are taken off, as is the liquid found there, together in the form of a suspension through the suspension discharge line 16. This causes a cocurrent or descending liquid stream through the holes 30 of the transverse false bottoms 28. The rate of withdrawal of the suspension of washed terephthalic acid particles, which, in this embodiment, is equal to the inflow rate of the fed suspension, is chosen, so that in the center part 22, including the separation stages, a velocity of the cocurrent stream prevails that is sufficient to fluidize the bed of terephthalic acid particles, which has formed in the vicinity of each false bottom in the preceding process step, and the fluidized particles are transported downward through the holes 30 of the false bottoms 28. Before a significant amount of the transported particles of terephthalic acid are transported through the next lower false bottoms, inflow of the supplied suspension and discharge of the suspension of washed terephthalic acid particles is interrupted by the timer switch 20, which causes activation of the control device 74 for the suspension feed valve and control device 80 for the suspension discharge valve, which closes the suspension feed valve 16 and the suspension discharge valve 72. Because of this, the entire liquid stream through the column is interrupted, and, as a result, it is possible for the terephthalic acid particles to settle under the influence of gravity within the entire tower until they reach the false bottom 28. In the vicinity of false bottom 28, they collect, agglomerate and form beds of non-fluidized solid particles. Solid particles that settle in the bottom space 26 of the scrubber collect in the vicinity of bottom 52. After a time period sufficient for significant buildup of beds, but not up to the overlying false

bottom, has elapsed in tower 10, the control device 76 for the wash liquid valve and the device 78 for the discharge valve of the outflowing liquid are activated by means of the timer switch 20, in order to open the wash liquid valve 68 and the discharge valve 70. Because of this, a liquid countercurrent from the sprinkler ring 62 to the inlet 42 of the discharge tube of the outflowing liquid 18 is produced. The flow rate is chosen so that, in the vicinity of each false bottom, a flow rate prevails that is sufficient to fluidize each of the beds, to displace less pure liquid by the pure liquid that is flowing through the holes 30, and to transport the terephthalic acid particles usually upward. After a sufficient time has elapsed in order to fluidize the beds and transport the particles upward without a significant amount of the particles flowing back again through the holes 30, through which they have already passed, the timer switch 20 causes the wash liquid valve control unit 76 to close the wash liquid valve 68 and, at the same time, causes the discharge valve control unit 78 to close the discharge valve 70 for the outflowing liquid. The entire liquid stream within scrubber 10 is therefore interrupted and settling of the terephthalic acid particles under the influence of gravity is made possible in the vicinity of each false bottom 28, where they again collect, agglomerate and form a non-fluidized bed. The sequence of steps is then repeated.

The time and flow rate values in the sequence of steps according to the embodiment just described are shown in Fig. 6 for a pilot unit of the scrubber 10, which is described by the following data:

Inside diameter of the central part 22 containing the separation stages - 10 cm,
Height of the central part 22 containing the separation stages = 1.5 m,
Inside diameter of the headspace 24 = 29 cm,
Height of the headspace 24 = 1.2 m,
Number of false bottoms 28 = 11,
Vertical spacing between false bottoms 28 = 8.4 cm, inside diameter of holes 30 = 3.1 mm.

At a spacing between the centers of the holes 30 of 6.2 mm and during operation with water as wash liquid at a temperature of about 43°C, the supplied suspension consists of an aqueous

mother liquid and terephthalic acid particles in a concentration of 19.3 weight percent, with the following particle size distribution:

Residue at a mesh size	US Screen Size	Weight percent (on a cumulative basis)
0.177 mm	+ 80 mesh	50.1
0.105 mm	+ 140 mesh	80.0
0.04 mm	+ 325 mesh	91.5

as well as an average particle size of 210 μm and an average settling rate of 63 cm/min in water at 40°C. Typical results under these conditions are an outflowing liquid with a solids content of 1.2 parts by weight, a suspension of washed terephthalic acid particles with a concentration of solid particles of 21.0 weight percent, a purification factor of 115, a washing efficiency of 99.2% and a solid throughput of 1549 $kg/m^2 \cdot h$.

Washing efficiency = [(weight of foreign substance content of the supplied suspension plus weight of the foreign substance content of the wash liquid) - (weight of the foreign substance content of the suspension of the washed terephthalic acid particles)] divided by (weight of the foreign substance content of the supplied suspension plus weight of the foreign substance content of the wash liquid).

Purification factor = concentration of the foreign substance content of the supplied suspension divided by the concentration of foreign substance content of the suspension of the washed terephthalic acid particles.

Solid throughput = amount per time (in kg/h) of the solid particles in the suspension of washed terephthalic acid particles that is removed from the tower, divided by the inside cross sectional area of the central part 22 containing the separation stages.

Fig. 7 shows another method of operation of the apparatus of Fig. 1, in which this method of operation corresponds to a preferred embodiment of the method according to the invention. In this operating method, the suspension feed valve 66 and the discharge valve 70 of the outflowing

liquid are opened without interruption during the entire sequence of steps. On completion of the settling period, the timer switch 20 causes the wash liquid valve control device 76 to open the wash liquid valve 68 to an extent and for a period so that the liquid countercurrent in the tower occurs with a rate and for a period sufficient to fluidize the beds of terephthalic acid particles in the vicinity of the false bottoms, and to displace the liquid from these beds in an upward direction, but not sufficient to transport the terephthalic acid particles upward through the holes 30 of the false bottom 28. At the end of the countercurrent step, the timer switch 20 causes the wash liquid valve control unit 76 to close the wash liquid valve 68. Settling of the terephthalic acid particles under the influence of gravity again occurs and the particles again collect, agglomerate and form a non-fluidized bed in the vicinity of each false bottom 28. At the end of the time period for this settling step under the influence of gravity, the timer switch 20 causes the wash liquid valve control unit 76 to open the wash liquid valve 68 to a predetermined flow rate and causes the valve control unit 80 to open the discharge valve 72 for the suspension. The flow rates of the wash liquid and the withdrawn suspension are chosen, so that they result in a cocurrent flow within the central part 22 containing the separation stages with a velocity sufficient to fluidize the corresponding particle bed in the vicinity of each false bottom 28, and to transport the particles through the holes 30 into the underlying stage, so that only minimal particle transport through the holes 30 of the underlying false bottom or bottoms occurs. This reduces the flow rate of withdrawn outflowing liquid. At the end of the period for this operating step, the timer switch 20 causes the wash liquid valve control device 76 to close the wash liquid valve 68, and causes the suspension discharge valve control unit 80 to close the suspension discharge valve 72. The sequence of steps is then repeated.

During displacement of liquid and washing of the solid at 8.9°C in scrubber 10 with the dimensions, etc. that were described in conjunction with Fig. 6, [with] the suspension of terephthalic acid particles in an aqueous mother liquor with a particle size distribution, as mentioned above, and a terephthalic acid particle concentration of 19.1 weight percent and with water as wash liquid, in which the flow rates and times of each step correspond to those in Fig. 7, and with a concentration of washed terephthalic acid particles in the suspension withdrawn from the scrubber 10 of 27 weight percent, a purification factor of 175, a washing efficiency of 99.6% and a solid throughput of 1580 $kg/m^2 \cdot h$ were obtained as typical results.

Fig. 8 shows another method of operation of the apparatus of Fig. 1 according to another embodiment of the method of the present invention. In this method of operation, the suspension feed valve 66 and the discharge valve 70 of the outflowing liquid are open during the entire sequence of steps. In addition, during the settling step under the influence of gravity, the wash liquid valve 68 is open to the extent that the flow rate of the resulting wash liquid countercurrent upward through the tower is smaller than the free settling rate of the smallest occurring particle size. At the end of the settling step, timer switch 20 causes the suspension discharge valve control device 80 to open the suspension discharge valve 72 and causes the wash liquid valve control device 76 to open the wash liquid valve 68, in order to further increase the flow rate of the wash liquid in the scrubber and, in so doing, equalize the discharge rate of the liquid taken off with the particles washed with the suspension, in which this equalization occurs to an extent that only the smallest possible amount, if any, of the terephthalic acid particles are transported by the cocurrent liquid stream through the central part 22 containing the separation stages through more than one false bottom 28. Because of removal of the suspension, a reduction in discharge rate of the discharged liquid through the liquid discharge pipe 18 is obtained. This sequence of steps is then repeated continuously.

In a scrubber 10 with the dimensions, etc., as described in conjunction with Fig. 6, during displacement of the liquid and washing of the solid at about 43°C with a suspension of terephthalic acid particles in an aqueous mother liquor, with a particle size distribution as described above and a terephthalic acid particle concentration of 16 weight percent, with water as wash liquid, in which the flow rate and time for each step is shown in Fig. 8, and with a concentration of washed terephthalic acid particles in the suspension removed from scrubber 10 of 26.5 weight percent, a purification factor of 200, a washing efficiency of 99.8% and a solid throughout of 2500 $kg/m^2 \cdot h$ was obtained as typical result.

The present invention therefore provides an improved method for the purification of crude or impure terephthalic acid. Not only are channeling of the wash liquid and back-mixing of the displaced liquid reduced to a minimum, but washing and scrubbing of the terephthalic acid particles by the wash liquid and displaced liquid are reinforced.

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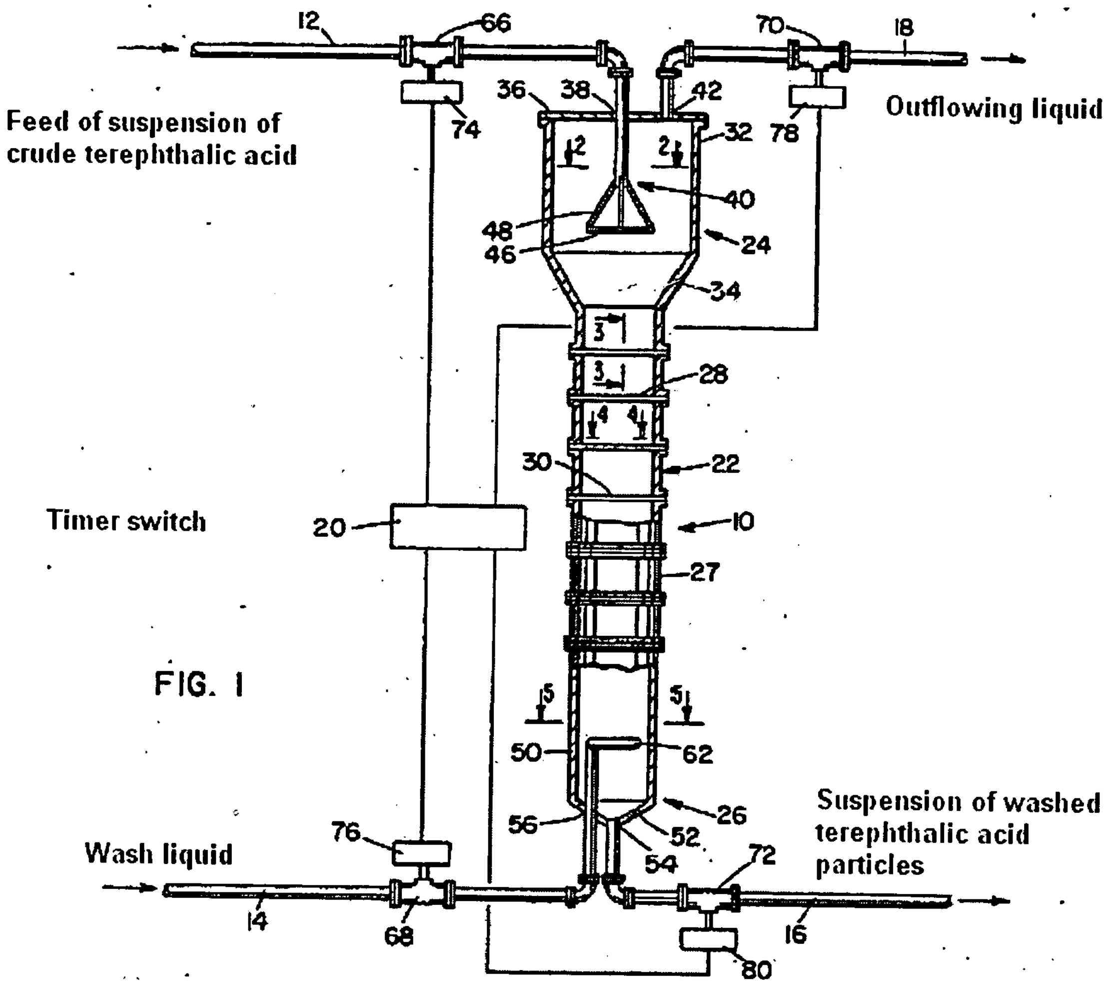

FIG. 1

FIG. 3

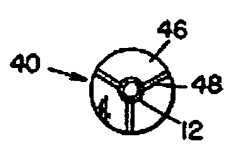

FIG. 2

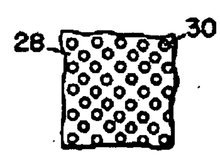

FIG. 4

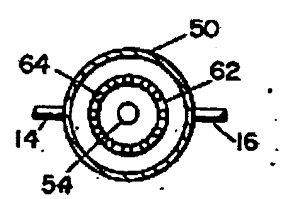

FIG. 5

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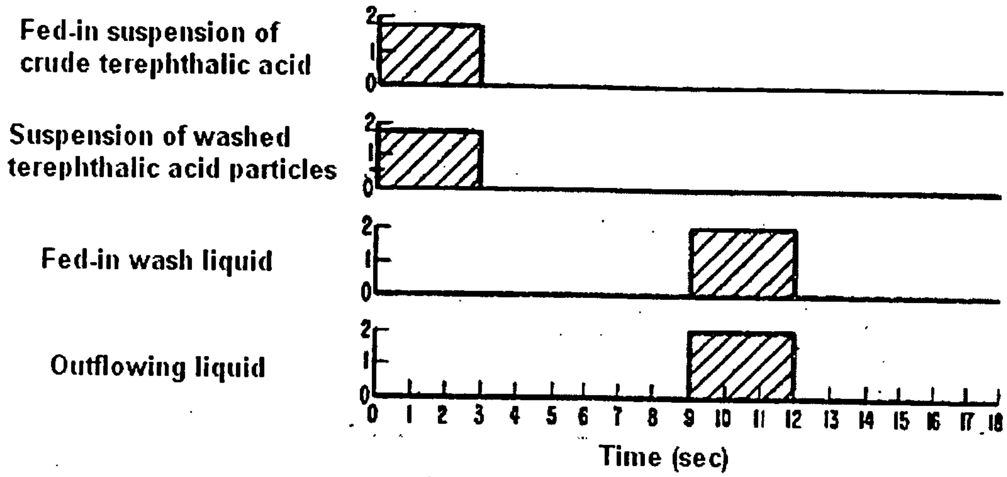

FIG. 6

1 GPM =
3,785 l/min

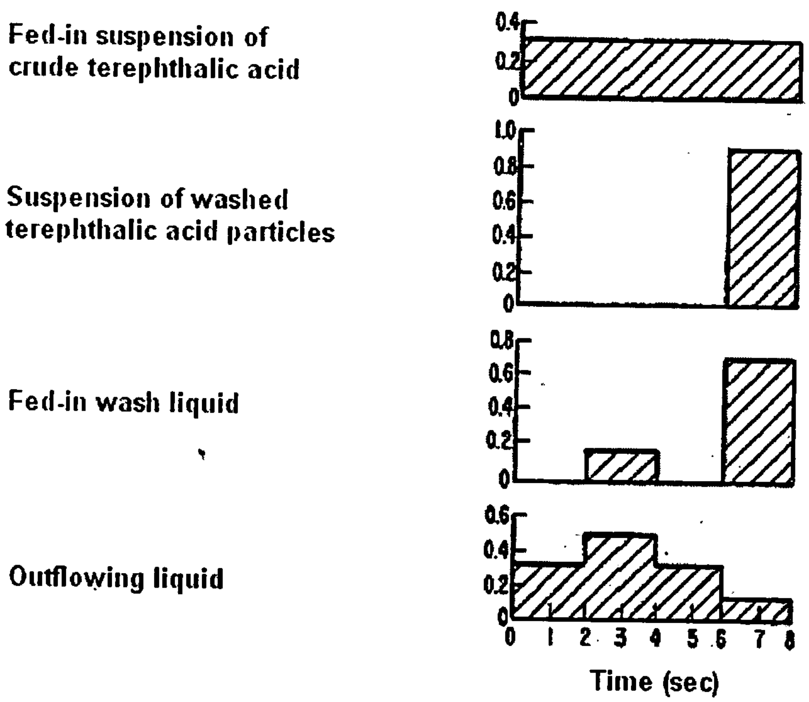

FIG. 7

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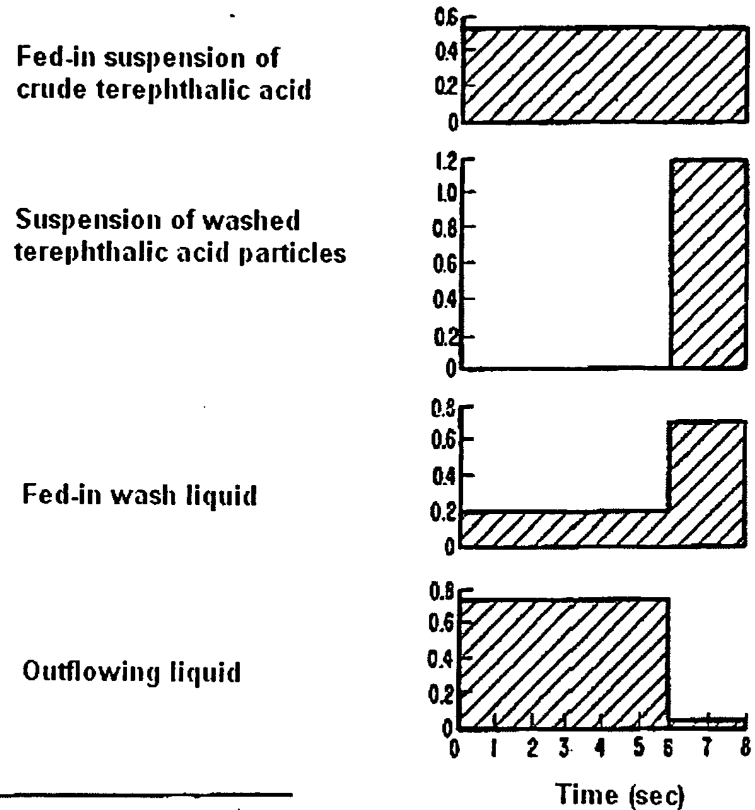

1 GPM = 3,785 l/min

FIG. 8

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